

The Effect of Oil Palm Waste Pyrolysis Bio-oil on Modified Bitumen Properties

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Abstract: The shortage and insufficient supply of asphalt binder has triggered the researcher to explore another alternative waste material to be blended with the binder. This study presents the performance of pyrolysis oil palm empty fruit bunch (EFB) bio-oil as an alternative binder in modified bitumen mixtures. In this study, the oil palm EFB was first pyrolyzed using auger pyrolyzer to extract the bio-oil. The PEN 80/100 bitumen was used in this study as a control sample whereas percentages of pyrolysis EFB bio-oil were varies from 5% and 10%. There are four bitumen tests that are performed in order to determine the properties of binder with the addition of EFB bio-oil, these include penetration, softening point, viscosity and dynamic shear rheometer (DSR). The findings showed that EFB bio-oil modified bitumen binder has improved rheological properties. The higher failure temperature for rutting resistance was achieved at 70°C which indicates low temperature susceptibility. The viscosity for modified bitumen decreased which indicates that it has lower friction and resistance to flow.

Introduction

Recently, the increase in the demand of crude oil due to the rising number of automobiles on the roads have led to a significant reduction in the crude oil reserves worldwide and in turn brought about the soaring in the price of the bitumen. This situation has drawn the attention of many experts or researchers to seek a more economically and environmentally sustainable ways through the investigation of the productions of alternative binders that can suit well for hot mix asphalt (HMA). Besides, this approaches can also produce a sustainable bio-binders that can lowered the mixing and compaction temperature of the asphalt mixtures as an attempts to reduce the heat emissions that mainly came from the asphalt industry which was believed to worsened the effects of global warming. The application of pyrolysis bio-oils for the modification of asphalt binders could reduce the compaction temperature, ageing and mixing time of the asphalt mixtures to a significant extend. In addition, it is also proven that the addition of bio-oils into the asphalt mixtures indicated a reduction in the rutting resistance and fatigue cracking as well as decreasing the dynamic modulus or the stiffness of the mixtures. On the other hand, there is also a significant improvement in the aspect of thermal cracking for the bio-modified asphalt binders.

Problem Statement. The gradually exhausting crude oil reserves worldwide has led to the concern of the many experts in the field of asphalt industry to explore the new alternative resources to reduce the dependency on the petroleum products as the road construction materials. Challenges are also faced by the contractors who involved in road construction industry as the price of crude oil experienced continuous fluctuation recently due to the low availability and high demand of the crude oil. The utilization of pyrolysis bio-binders derived from the biomass could offer a cheaper and easily available materials for the existing asphalt industry to deal with the current issues and problems related to the availability and price of crude oil. The conventional asphalt binders also has lower fatigue cracking and rutting resistance as well as faster ageing when compared with the bio-binders. These properties has led them to be the unsuitable road material to cater for the increasing traffic loading on the road surface due to the soaring of traffic volume nowadays. The application of bio-modified asphalt can give better improvements to the performance of the road layers so that they are able to withstand high temperature and moisture level without any defects in the long term.

Objective of study The aim of this study is to investigate the suitability of fast pyrolysis bio-oils for the modification of asphalt binders that can suit well as the pavement materials. The specific objectives of this study are as follows:

- 1) To convert oil palm EFB into bio-oil through pyrolysis method
- 2) To investigate and compare the properties of the asphalt mixtures modified by bio-oil derived from oil palm wastes with that of the conventional asphalt binders.

Scope of study This study focuses on the laboratory investigations on the effectiveness of the addition of bio-oil for the modification of bitumen as a way to improve its properties and performance grade.

The pyrolysis process will be conducted to extract the bio-oil from oil palm empty fruit bunch. The palm oil wastes are chosen as so far not much study has done on the extraction of its bio-oil for the purpose of application of green concept in asphalt industry. The extracted bio-oil is used to modify the conventional asphalt binder in the percentage of 5% and 10% respectively to study differences that will bring about by the percentage difference in the amount of pyrolysis bio-oil added for the modification process.

Laboratory testing which includes Penetration Test, Softening Point, Viscosity and Dynamic Shear Rheometer (DSR) are parts of the study to investigate the behavior of the bio-modified asphalt binders as an approach to evaluate its physical and rheological properties as compared to the conventional asphalt binder.

Previous study

It is commonly known that conventional bituminous materials performed their function satisfactorily in most of the pavements. However, existing highway systems nowadays have been dealing with increased traffic volume, higher axle load and extreme environmental impacts such as acid rain and high surrounding temperature. The situation is evident for the last three decades, that the pavement has been facing more demands than before resulting in the need for an enhancement in the properties of bituminous materials [1].

Many studies were done in the past to evaluate the effect of the addition of bio-oils extracted from agricultural wastes such as oakwood, switchgrass, palm kernel shell and swine manure on the rheological properties, low temperature performance as well as the fatigue cracking and rutting resistance of the bio-modified asphalt binders.

The approaches to produce bio-based asphalt binders due to the rising price of crude oils and environmental issues have drawn the attention of many researchers for decades. In fact, replacement or blending of the existing asphalt with the bio-oils produced from pyrolysis of the crop wastes are commonly used. Bio-modified asphalt binders are the better choice over the virgin asphalt binder as a paving material because of its durability characteristics. Table 1, summarize previous research work.

Table 1: Summary of previous research work

No.	Author	Research Title	Performance
1.	[2]	Performance Evaluation of Asphalt Binder Modified by Bio-oil Generated from Waste Wood Resources	<p>DSR -Addition of pyrolysis bio-oil into virgin asphalt improved high temperature performance</p> <p>Viscosity -Lower the mixing temperature of asphalt mixtures with addition of bio-oil</p> <p>Penetration Index - Original bio-oil (OB) modified asphalt binders demonstrated the lowest stiffness - Dewatered bio-oil (DWB) and polymer modified bio-oil asphalt binders showed moderate and highest stiffness respectively</p>
2.	[3]	Laboratory Evaluation of Asphalt Mixtures Containing Bio-Binder Technologies	<p>DSR</p> <p>Rutting Resistance - Bio-modified asphalt mixtures possessed improved rutting resistance performance as compared to the conventional asphalt binders</p> <p>Fatigue Cracking Resistance - Bio-Asphalt mixtures have improved low temperature fracture performance and a tensile strength of more than 80% when moisture content was taken into consideration</p>
3.	[4]	Aging Influence on Rheology Properties of Petroleum-Based Asphalt Modified with Biobinder	<p>Viscosity - Viscosity value decrease with increasing bio-oil content</p> <p>DSR</p> <p>Rutting Resistance ($G^*/\text{Sin } \delta$) - Increase in high temperature rutting resistance performance as the concentration of bio-oil increase</p> <p>Fatigue Cracking Resistance ($G^* \text{ Sin } \delta$) - Pyrolysis bio-oil modified asphalt binders have improved low temperature performance in terms of cracking properties</p>
4.	[5]	Rheological Properties of Polyacrylates used as Synthetic Road Binders	<p>DSR</p> <p>Rheological Properties - Similarities in rheological properties between conventional and bio-oil modified asphalt which are unaffected by their differences in term of temperature susceptibility - Synthetic binders showed almost the same rheological properties with polymer modified bitumen in term of ability to switch between viscous and elastic dominated behavior</p>

			<p>Penetration</p> <ul style="list-style-type: none"> - Bio-binders exhibit soft behavior and is more suitable to be used as modifier for hard asphalt rather than as a replacement
5.	[6]	Utilization of Fractionated Bio Oil in Asphalt	<p>DSR</p> <ul style="list-style-type: none"> - Biomass-derived bio-oils can give improvement to the performance grade of bio-asphalt mixtures by nearly 6°C - Significant upgrading in performance grade of the bio-asphalt binders can be witnessed with the blending of pyrolysis bio-oils as much as 9%
6.	[7]	Development of Non-Petroleum Based Binders for Use in Flexible Pavement	<p>Viscosity</p> <ul style="list-style-type: none"> - Viscosity of bio-binders were studied by using Arrhenius-type model which found that viscosity follows an exponential relationship with the temperature - Increase in temperature will lead to exponential decrease of the viscosity of bio-binders

Methodology

The purpose of this study was to investigate the effects of the modification of bitumen with bio-oil on the properties of the modified bitumen. This chapter discusses several tests that will be conducted to achieve the objectives of the study. Study will be carried out by using experimental methods to evaluate the quality of bio-oil and its suitability in road pavement especially in hot mix asphalt. Laboratory experiment is done to identify the actual performance of the bio-oil and its suitability by comparing to normal bitumen based on the standard specification of JKR/SPJ/1988, ASTM and AASHTO.

Process Framework

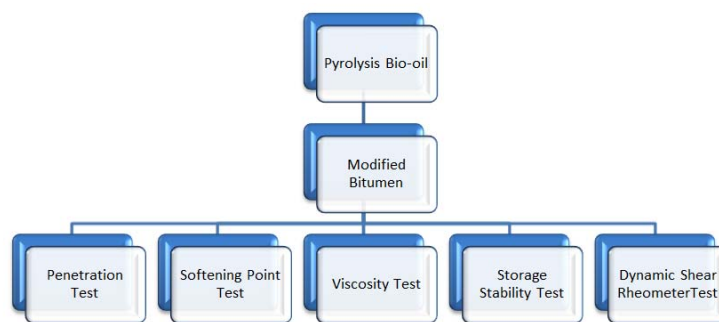


Figure 1:Flow chart of research testing operation

Bio-oil Extraction The bio-oil extraction process is conducted by using auger pyrolyzer to heat the empty fruit bunch (EFB) in order to collect pyrolysis bio-oil as the end product. In this process, the EFB is first dried thoroughly in the oven for 24 hours. Prior to the commencement of the fast pyrolysis process, the auger pyrolyzer is set with an operating temperature 700°C and the auger's rotating revolution of 18 rpm while the condenser is set at a temperature of 5°C to condense the syngas emitted from the heating of the EFB into droplets of bio-oil. The pressure meter of nitrogen gas container is adjusted to have a pressure of 16 psi to avoid the pyrolyzer from being burned at high operating temperature. The EFB is then poured into auger pyrolyzer at its inlet. Each time only

50g of EFB are allowed as the amount more than that can easily lead to clogging of the instrument. The bio-oil is collected at the bottom of the condenser by using a small beaker. The process is repeated until the desired amount of bio-oil is extracted.

Sample Preparation For the sample preparation, bitumen with penetration grade 80-100 will be used in this study. The materials used are bitumen and bio-oil which act as a modifier. The blending of bio-oil is done at a percentage of 5% and 10% into the pre-heated virgin bitumen at a temperature of 140°C. The process of the preparation of modified bitumen specimens starts with the weighing of both the bitumen control specimen and the pyrolysis bio-oil according to the quantity needed. The bitumen specimens are then placed into an oven at a temperature of 140°C so that it changes into liquid phase. 5% and 10% of the pyrolysis bio-oil are prepared by weight of the specified amount of bitumen control specimen respectively. The pyrolysis bio-oil is then slowly added into the heated bitumen specimens. Always make sure that the temperature of the mixture does not exceed 140°C to avoid the bitumen from getting burned. The mixture is blended properly by using high shear mixer at a constant speed of 1000 rpm for 1 hour at 140°C until it is uniformly mixed.

Bitumen Laboratory Tests Penetration Test Penetration test is performed to determine the consistency of the modified bitumen sample by determining the distance in tenths of a millimeter that a standard needle penetrated vertically into a sample of the material under fixed conditions of temperature, load and time. The heated bitumen sample is first poured into the sample container to a depth such that, when cooled to the temperature of test, the depth of the sample is at least 10mm greater than the depth to which the needle is expected to penetrate. It is then allowed to cool in the atmosphere at a temperature of 25°C for one hour. The penetration needle is positioned by slowly lowering it until its tip just makes contact with the surface of the sample. The needle holder is then released and the penetration reading is taken from the penetrometer dial. Five determinations are made at points on the surface of the sample not less than 10mm from the side of the container and not less than 10mm apart. Before repeating the test, the penetration needle is cleaned with toluene and dried by using a clean cloth.



Figure 2: Penetration test

Softening Point Test Softening point test is conducted to determine the temperature of asphalt binder within the range between 30°C and 157°C by means of the ring and ball apparatus as shown in Figure 3. In this test, the heated modified bitumen sample is poured into rings placed on aluminium foil apply with soap to give an excess above the top of the ring when cooled. The rings are then to cool at room temperature. The excess sample is removed with warm blade so that the test specimens are leveled with the top of the rings. The appropriate thermometer is inserted through the hole in the top plate and adjusted so that the bottom of the thermometer bulb will be level with the bottom of the rings, i.e. just below (0.4 mm) the bottom of the ring holder. The filled rings are placed in the ring holder while the ball-centering guide is positioned on the rings. With the distilled water in the bath

at $5 \pm 2^{\circ}\text{C}$, the assembled apparatus and the steel balls are transferred to the bath. The level of the water is ensured to be 50 ± 5 mm above the tops of the rings. The bath temperature is maintained at $5 \pm 2^{\circ}\text{C}$ for 15 min, then using tongs to place a steel ball on each specimen. Heat is applied to the bath and the bath liquid is stirred so that the temperature of the bath liquid rises at $5 \pm 0.5^{\circ}\text{C}$ per min. For each test specimen, estimate to the nearest half division the temperature shown by the thermometer at the instant that the asphalt sample surrounding the ball touches the base plate. The temperature at which the ball touches the plate is recorded.

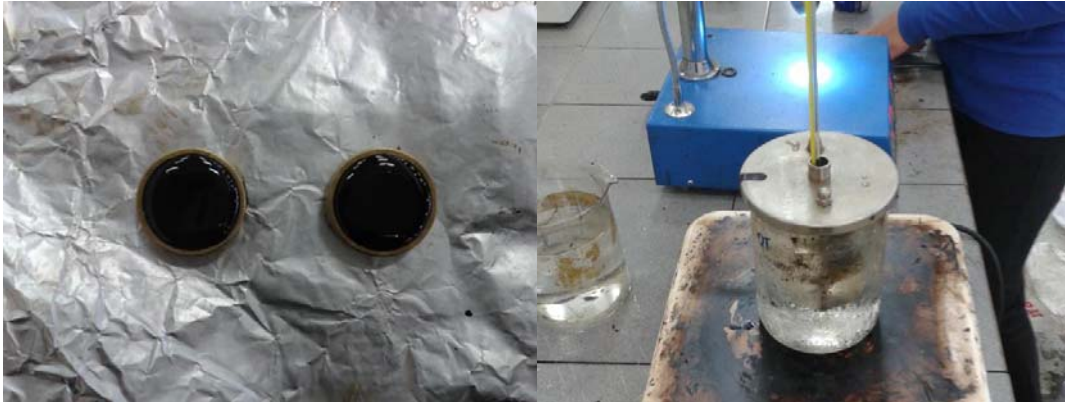


Figure 3: Softening point test

Dynamic Shear Rheometer Test Dynamic shear rheometer (DSR) involves a parallel plate rheometer that is used to apply shear strain or shear stress under a controlled temperature and frequency. It is used to characterize the viscous and elastic behavior of asphalt at high and intermediate service temperatures which can evaluate rutting and fatigue cracking potential of pavement. In this test, the asphalt binder from which the test specimens are to be selected is heated until the binder is sufficiently fluid to pour the test specimens. The DSR is then heated to the test temperature. After that, the asphalt binder sample is placed between the test plates. The test plates are moved together until the gap between them equals the test gap plus 0.05mm. The specimen is trimmed around the edge of the test plates using a heated trimming tool. The test plates are moved together to the desired testing gap. This creates a slight bulge in the asphalt binder specimen's perimeter. The specimen is then brought to the test temperature. The test is started after the specimen has been at the desired temperature for at least 10 minutes. The specimen in the DSR is conditioned for 10 cycles at a frequency of 10 rad/sec (1.59 Hz). The test measurements are taken by DSR over the next 10 cycles and then the software reduces the data to produce a value for complex modulus (G^*) and phase angle (δ).

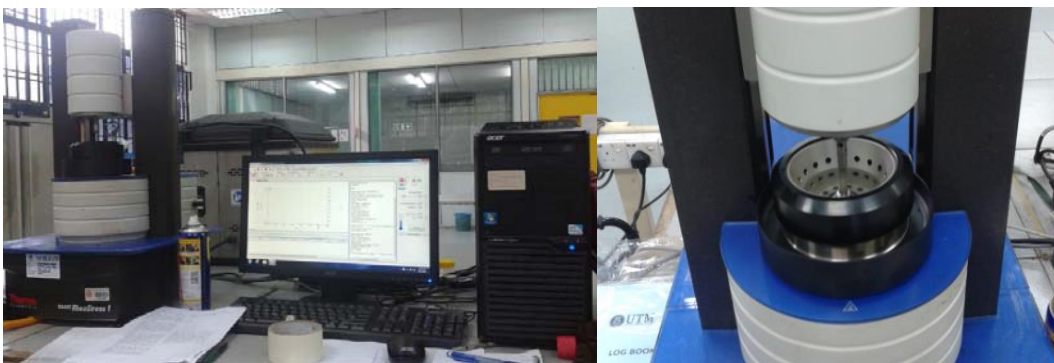


Figure 4: Dynamic shear rheometer (DSR) test

Viscosity Test This test is conducted via rotational viscometer to determine the resistance to flow of bitumen. The rotational viscometer senses torque required to rotate a spindle at constant speed while immersed in the modified bitumen sample. The desired dynamic viscosity value which is proportional to this measured torque will eventually be obtained. When conducting the test, the spindle, sample chamber, and viscometer environmental chamber (Thermosel) are first preheated to 135°C. The modified bitumen sample is then heated until fluid enough to pour. The sample is stirred carefully to make sure it is not entrapping air bubbles. Appropriate amount of asphalt binder is then poured into sample chamber. The sample chamber is inserted into RV temperature controller unit and spindle is lowered carefully into the bitumen sample. The bitumen sample is then brought to the test temperature of 135°C within approximately 30 minutes and allowed to equilibrate at test temperature for 10 minutes. After that, the spindle is adjusted to be rotated at 20 RPM, making sure the percent torque as indicated by the RV readout remains between 2 and 98 percent. Once the sample has reached temperature and equilibrated, 3 viscosity readings are taken from RV display, allowing 1 minute between each reading. Viscosity is reported as the average of 3 readings. The test is then repeated with test temperature of 165°C.



Data Analysis

Penetration Test Results In this study, penetration test has been conducted for every sample and the results are then compared with the virgin asphalt (80/100 PEN). Three different samples, i.e. samples with 0%, 5% and 10% bio-oil were tested using a standard needle loaded with 100 g of loads and the readings were noted from the penetrometer dial. Based on the Figure 5, it can be seen that the EFB bio-oil can be added up to 5% to achieve the grade of 80/100 PEN at the value of 90.5 which is comparable with that of the virgin bitumen at the value of 86.9. For the addition of 10% bio-oil into bitumen, it will significantly increase the penetration value up to a maximum penetration value of 112.4. This shows that the modified bitumen becomes softer as more percentage of EFB bio-oil is blended with the bitumen. This result is supported by the previous research conducted by [2] which proved that original bio-oil modified asphalt exhibits low stiffness. This property would be useful to obtain a softer modified bitumen binder through the blending of the EFB bio-oil into harder grade bitumen, which is suitable to be used in cold climate region whereby softer bitumen is much more preferred. The application mentioned complies with the results proven by the study done by [1] which indicated bio-binders exhibit soft behaviour and is more suitable to be used as modifier for hard bitumen rather than as a replacement.

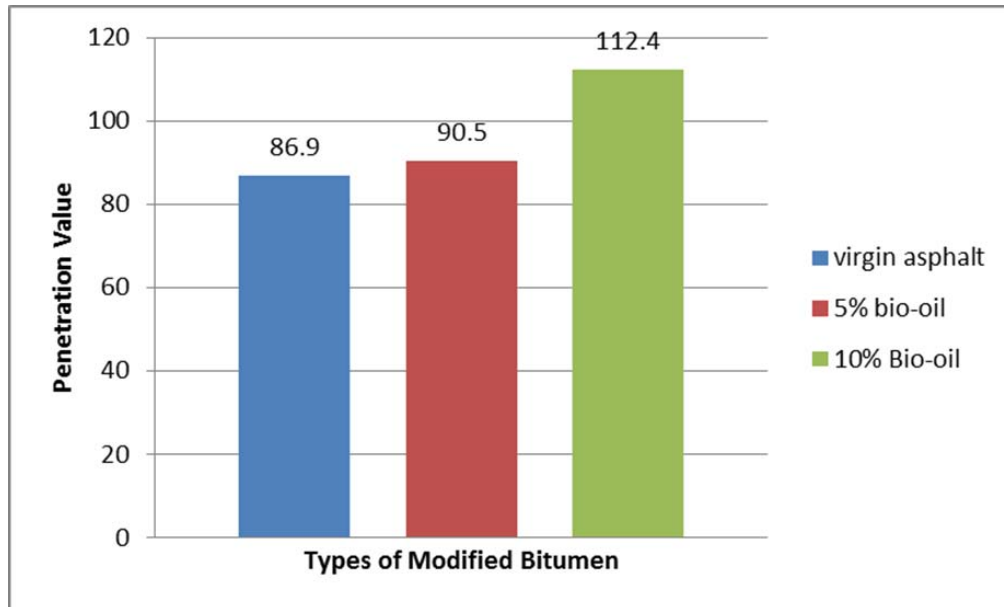


Figure 5: Penetration value for different percentage of bio-oil content

Softening Point Results Softening point tests were done for normal bitumen and modified bitumen with 5% and 10% of EFB bio-oil. The average softening point values of modified bitumen were then compared with temperature value of normal bitumen (80/100 PEN). Figure 6 shows the softening point test results for different percentage of EFB bio-oil used as modifier for bitumen binder. In general, the higher the EFB bio-oil content, the lower the softening point. From the results, it can be observed that the control virgin bitumen sample has the highest softening point, which is 41°C. The softening point for modified bitumen with 5% of EFB bio-oil as modifier decline 3°C from the softening point of the control bitumen sample to a value of 38°C, whereas the softening point of modified bitumen with 10% bio-oil modifier decline significantly to a value of 25.2°C, which is the lowest among the three bitumen samples. The findings from the softening point tests indicated that the bitumen become more susceptible to temperature change as EFB bio-oil content increased.

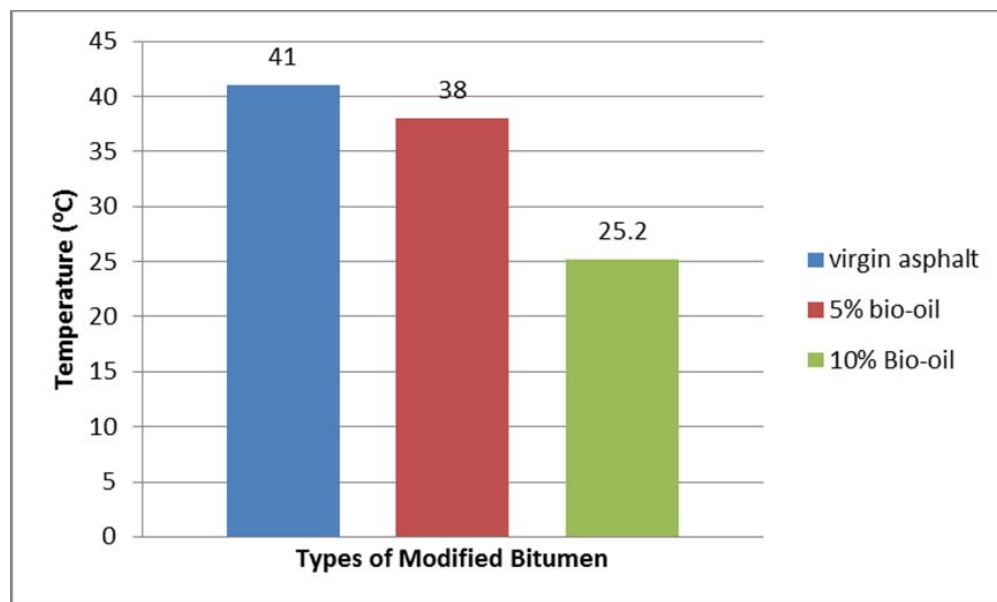


Figure 6: Softening Point Value for different percentage of bio-oil content

Penetration Index The PI value describes the modulus or stiffness of bitumen at any loading time and temperature. According to the asphaltic materials classification table, PI value in the range of -2 to +2 replicates the conventional paving bitumen. Table 2 shows the results of penetration index for all samples determined from the nomograph using the penetration and softening point values. From the table, it can be observed that bitumen binder samples blended with EFB bio-oil have the PI values which are less than -2. The bitumen samples with 5% and 10% of EFB bio-oil as modifier have PI values of -3.53 and -9.86 respectively, which showed that they are considered to be susceptible to temperature changes.

Table 2: Penetration Index (PI) for modified asphalt binder

Sample (% Bio-oil)	PEN value (dmm)	Softening Point (°C)	PI
0%	86.9	41.0	-2.52
5%	90.5	38.0	-3.53
10%	112.4	25.2	-9.86

Rutting Resistance Dynamic Shear Rheometer Test was conducted to obtain the rutting resistance of bitumen binder at which $G^*/\sin \delta$ is greater than 1 kPa. Based on Superpave specifications, $G^*/\sin \delta$ obtained from DSR test is used as a key factor to define the permanent temperature. Figure 7 shows the results of rutting resistance for all the modified bitumen binder.

The result indicates the maximum value of $G^*/\sin \delta$ is measured for virgin bitumen at temperature 46°C with the value of 28.6 kPa, followed by modified bitumen with 10% bio-oil at 23.04 kPa and the minimum rutting resistance value 11.48 kPa for modified bitumen with 5% bio-oil. It can be seen that the rutting resistance value was incrementally decreased as the test temperature increased. It is also observed that the $G^*/\sin \delta$ value for the bitumen increases as the percentage of EFB bio-oil increases.

This result complies with the previous research carried out by [2] who proved that addition of pyrolysis bio-oil into virgin asphalt improved its high temperature performance. Bio-modified asphalt mixtures possessed improved rutting resistance performance as compared to the conventional asphalt binders [3]. The increase in concentration of bio-oil will lead to increase in high temperature rutting resistance performance [4].

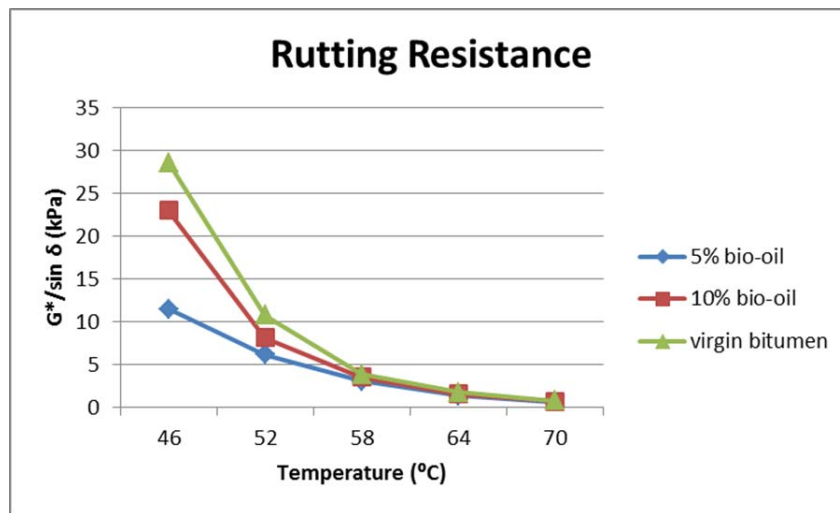


Figure 7: Rutting Resistance of modified bitumen with EFB bio-oil

High Failure Temperature The high failure temperature values from the DSR test gives indication that the binders are less susceptible to permanent deformation at high pavement temperature [8]. Figure 8 shows the high temperature failure results of bitumen binder modified with EFB bio-oil. The figure shows that the virgin bitumen and modified bitumen with 5% and 10% bio-oil have the same failure temperature, i.e. 70°C. This shows that the addition of bio-oil does not have much effect on the failure temperature of modified bitumen which in turn brings little influence to its permanent deformation properties.

This implies that the low temperature susceptibility and the stiffness to resist rutting deformation of the control bitumen 80/100 remain almost unchanged after the addition of EFB bio-oil as modifier.

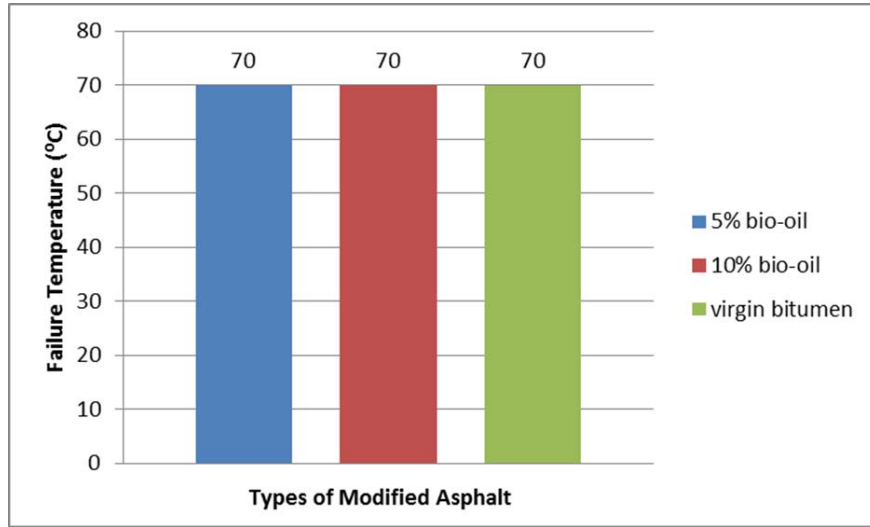


Figure 8: High failure temperature of asphalt binder with different percentages of bio-oil

Viscosity The rotational viscosity test result at temperature 135°C and 165°C on different percentages of bio-oil modified bitumen are shown in Figure 9 and Figure 10. The viscosity graph has depicted decreasing trends of viscosity value for modified bitumen binder with the addition of 5% and 10% of pyrolysis EFB bio-oil. At temperature 135°C, the virgin bitumen 80/100 shows highest viscosity value which is 0.467 Pa.s whereas the modified bitumen with 5% and 10% EFB bio-oil as modifier demonstrated same viscosity value of 0.40 Pa.s. From the result, it can be seen that the increase in bio-oil content will lead to the decrease in viscosity of bitumen samples. This result obeyed the research conducted by Mills-Beale, which proved that viscosity value of bitumen sample decrease with increasing bio-oil content. This implied that the bio-oil modified bitumen has lower resistance to flow and internal friction that exist in the sample. This is because the EFB bio-oil is less viscous and thus acts as lubricants which contributed to the low internal friction. The reducing viscosity value is related to the low adhesive performance of bitumen binder when high amount of EFB bio-oil is added in the asphalt binder [9] At temperature 165°C, the virgin bitumen 80/100 remains the highest viscosity of 0.133 Pa.s while both the bitumen sample of 5% and 10% EFB bio-oil content have the lowest viscosity value of 0.10 Pa.s. This implies that as the temperature increase, the viscosity of the virgin and modified bitumen samples will decrease. Viscosity follows an exponential relationship with the temperature as increase in temperature will lead to exponential decrease of the viscosity of bio-binders [7]. The addition of bio-oil as modifier will lower the mixing temperature of asphalt mixtures [2]. In economic aspect, low viscosity is favorable due as it can decrease the mixing and compaction temperatures of bituminous mixtures and hence leads to a more economical road construction which can save cost.

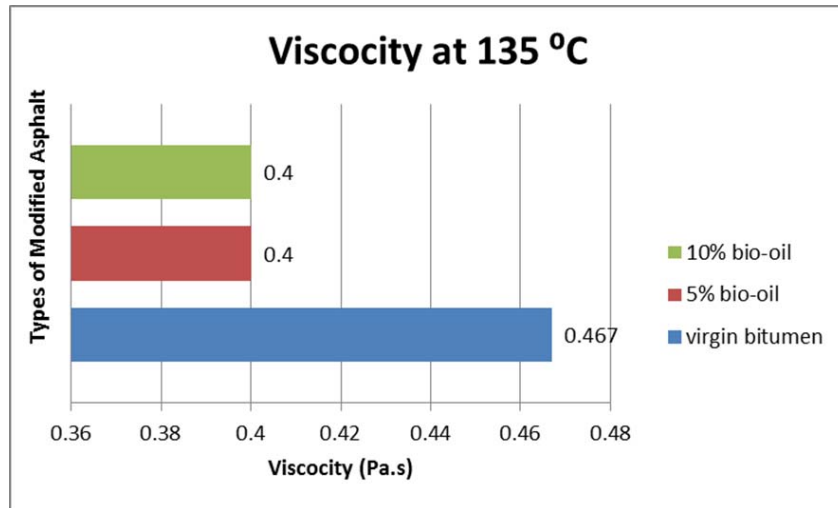


Figure 9:Viscosity at temperature 135°C

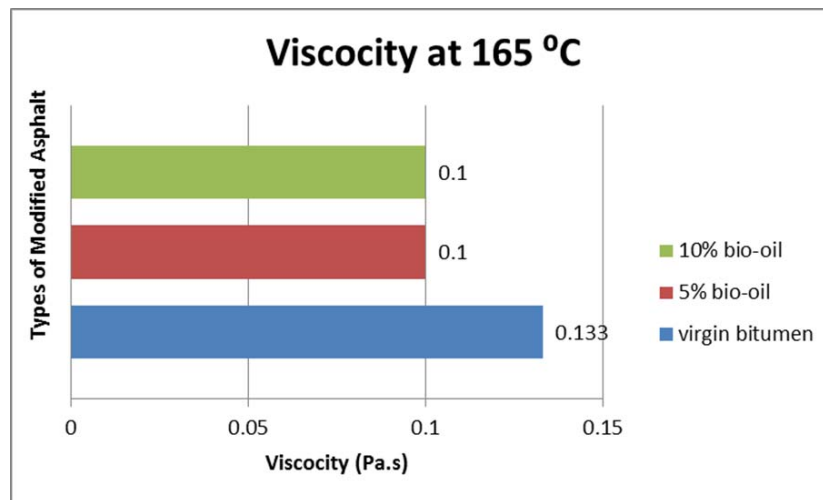


Figure 10:Viscosity at temperature 165°C

Conclusion

In general, the finding concludes that Modified bitumen tends to become softer and more susceptible to temperature change as more percentage of pyrolysis EFB bio-oil is blended with the bitumen. According to the PI values, all the modified bitumen binder samples are considered in the classification of temperature susceptible bitumen as their PI values are less than -2. The high temperature rutting resistance performance of modified bitumen increases with increasing pyrolysis EFB bio-oil content. Meanwhile, the viscosity value of bitumen sample decrease as more percentage of EFB bio-oil is blended which indicates the sample has lower internal friction and resistance to flow.

For future research, it is suggested that more sets of percentage of bio-oil with small percentage intervals have to be carried out so that the change in properties of the modified bitumen can be study in a more detail manners. It is also suggested that replacement of EFB bio-oil should be used instead of blending it as additive when producing bio-oil modified bitumen. Hot mix asphalt should be designed by using the EFB bio-oil modified bitumen through Marshall Mix Design method and then tested under Marshall Stability and Flow test to study the practicability of the modified bitumen in real situation of road construction. Bitumen grade 60/700 is recommended to be used

instead of bitumen grade 80/100 as the former is harder and more practical to be blended with pyrolysis EFB bio-oil which can give it softer behavior for application in cold climate region. The auger pyrolyzer should be checked against leakage so that the EFB bio-oil would not be lost as vapor during the pyrolysis process which can significantly affect the amount of bio-oil yields in the process.

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